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### Recommended Citation

Gutierrez, M.A.; Haselkorn, M.; Iglesias, P. The Lubrication Ability of Ionic Liquids as Additives for Wind Turbine Gearboxes Oils. *Lubricants* 2016, 4, 14. doi:10.3390/lubricants4020014

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## Article

# The Lubrication Ability of Ionic Liquids as Additives for Wind Turbine Gearboxes Oils

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Academic Editors: Antolin Hernández Battez and Rubén González Rodríguez

Received: 22 March 2016; Accepted: 22 April 2016; Published: 5 May 2016

**Abstract:** The amount of energy that can be gained from the wind is unlimited, unlike current energy sources such as fossil and coal. While there is an important push in the use of wind energy, gears and bearing components of the turbines often fail due to contact fatigue, causing costly repairs and downtime. The objective of this work is to investigate the potential tribological benefits of two phosphonium-based ionic liquids (ILs) as additives to a synthetic lubricant without additives and to a fully formulated and commercially available wind turbine oil. In this work, AISI 52100 steel disks were tested in a ball-on-flat reciprocating tribometer against AISI 440C steel balls. Surface finish also affects the tribological properties of gear surfaces. In order to understand the combined effect of using the ILs with surface finish, two surface finishes were also used in this study. Adding ILs to the commercial available or synthetic lubricant reduced the wear scar diameter for both surface finishes. This decrease was particularly important for trihexyltetradecylphosphonium bis(trifluoromethylsulfonyl) amide, where a wear reduction of the steel disk around 20% and 23% is reached when 5 wt % of this IL is added to the commercially available lubricant and to the synthetic lubricant without additives, respectively.

**Keywords:** ionic liquids; wear; lubrication; steel-steel contact

## 1. Introduction

Wind is a source of clean and sustainable energy. Wind energy does not produce harmful pollution gases such as carbon dioxide, sulfur dioxide, and other gases that have contributed to global warming. In addition, wind is a renewable source of energy, and a good alternative to current limited fossil fuel-generated electricity. While there is a big push in the use of wind energy, there is a downside regarding the cost of maintaining the wind turbines, more specifically the gearboxes [1,2]. Gears and bearing components often fail due to contact fatigue, causing costly repairs and downtime of the turbines [2]. The high cost of maintaining both land-based and off-shore wind turbines is a critical aspect of lowering the cost of wind energy and achieving the US Department of Energy (DOE) goal of generating 20% of the nation's electricity from wind energy in 2030 [3]. Several lines of action can be followed to achieve this goal. Improving the overall gearbox design [4–6] and using surface treatments [2] or coatings [7] to increase the wear resistance of the materials used for the contacting components are two of the most common ways investigated to reduce the maintenance costs of the turbines and increase the overall reliability and efficiency of wind technology.

In the last decade, the use of ionic liquids (ILs) has attracted attention in the tribology community as potential lubricants and lubricants additives for challenging contacts [8–11]. ILs, also known as fused salts and molten salts, are ionic compounds with melting points lower than 100 °C. They are typically

composed of bulky asymmetric organic cations and a weakly coordinating anion. Their unique properties, including high thermal stability, non-volatility, non-flammability, high ionic conductivity, wide electrochemical window, and miscibility with organic compounds make them ideal candidates for many engineering applications. In addition, ILs have the ability to form stable ordered layers and protective tribo-films [12–14] in the area between the two materials in contact, reducing friction and wear.

Some researchers [15,16] have shown that the addition of small amounts of halogenated ILs to mineral and synthetic wind turbine oils has a positive effect on the wear behavior of materials in contact. However, halogen-containing anions are very sensitive to moisture and, in the presence of water, will react, causing: (1) the corrosion of metallic surfaces by formation of metallic halides and (2) the evolution of toxic species (HF) from hydrolysis of the anions [17,18]. Recent literature has suggested the potential of using ILs with halogen-free anions [11,19,20]; however, studies on the tribological performance of these ILs as additives of lubricants for wind turbine applications are still very scarce.

The objective of this work was to investigate the potential tribological benefits of the use of two phosphonium-based ILs (one of which is halogen-free) as additives of lubricants, in combination with two surface finishes, on gearboxes of both land-based and off-shore wind turbines. The ILs were added to a synthetic oil without additives and to a fully formulated and commercially available wind turbine lubricant. The effect of the IL concentration was also studied and compared to the currently available gearbox oil.

## 2. Experimental

AISI 52100 steel disks were tested in a ball-on-flat reciprocating (Figure 1) tribometer against AISI 440C steel. Two different surface finishes (super-finish,  $R_a \approx 0.02 \mu\text{m}$  and normal ground,  $R_a \approx 0.1 \mu\text{m}$ ) were used for the disks. Table 1 summarizes the properties and dimensions of the materials used in this study. The following experimental parameters were kept constant for all tests: normal load = 14.7 N (corresponding to mean Hertzian contact pressure = 1.03 GPa and maximum Hertzian contact pressure = 1.55 GPa), frequency = 50 Hz, amplitude = 0.8 mm, sliding distance = 288 m, temperature = 23 °C, and relative humidity = 30%–35%.

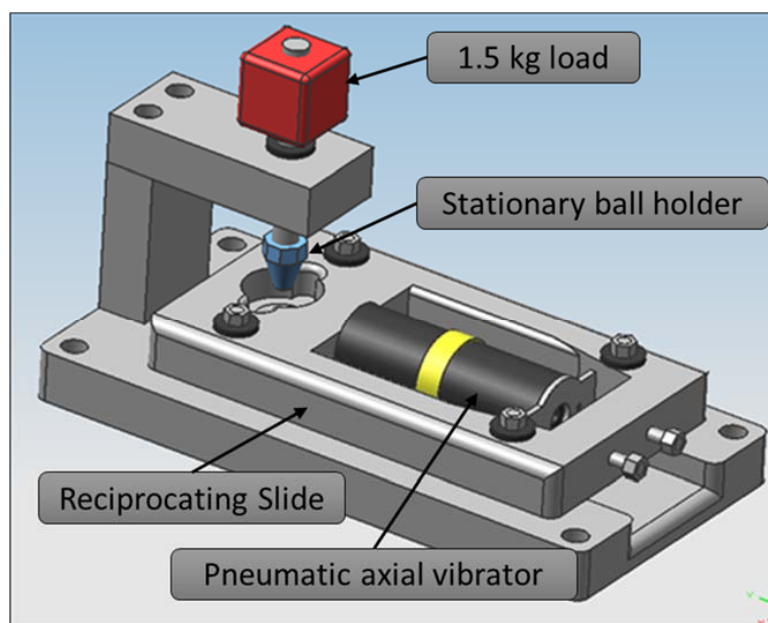


Figure 1. Ball-on-flat tester with reciprocating motion.

**Table 1.** Properties and dimensions of the materials used in this study.

	Disk	Ball
Material	AISI 52100 steel	AISI 440C
Hardness (HV)	500	690
Dimension (mm)	Diameter = 19 Thickness = 7	Spherical radius = 3

ILs used in this study were commercially available from Sigma-Aldrich (USA). Their molecular structure, name, and abbreviation (code) are shown in Table 2. Both ILs have identical cations with varying anion structures. Lubricating mixtures were prepared by adding 2.5 or 5 wt % ratio of ILs to a polyalphaolefin—Synton PAO-40 (PAO)—base stock and to a commercially available, fully formulated, wind turbine gearbox lubricant—Mobilgear SHC XMP 320 (MG). Before each test, steel disks were covered with 2 mL of the lubricant, and no additional lubricant was added during the test. Optical micrographs of wear track were obtained using a Zeiss 3-D stereoscope, and SEM images were obtained using an AMRAY 1830 scanning electron microscope with energy-dispersive X-ray spectroscopy (EDS) capability.

**Table 2.** Molecular structure, name, and abbreviation of ionic liquids (ILs) used in this study.

Code	Cation	Structure	Anion	IUPAC name
[THTDP][Deca]	$\text{H}_3\text{C}(\text{H}_2\text{C})_5-\text{P}^+-\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2-$	$(\text{CH}_2)_5\text{CH}_3$	$\text{H}_3\text{C}(\text{H}_2\text{C})_8-\text{C}(=\text{O})\text{O}^-$	Trihexyltetradecylphosphonium decanoate
[THTDP][NTf2]	$\text{H}_3\text{C}(\text{H}_2\text{C})_5-\text{P}^+-\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2-$	$(\text{CH}_2)_5\text{CH}_3$	$\text{F}_3\text{CO}_2\text{S}^--\text{N}^+(\text{SO}_2\text{CF}_3)_2$	Trihexyltetradecylphosphonium bis(trifluoromethylsulfonyl) amide

Upon completion of a test, the wear ball and disk were removed from the tribometer and cleaned with isopropyl alcohol. The major and minor axes of the wear scar were measured according to ASTM-D6079. Three tests were run for each lubricant mixture, and the reported value is the average value of the three tests.

Densities at room temperature were determined by weighing a known volume. Viscosities at 40 °C and 100 °C were obtained using a LVDV2T Brookfield viscometer with a Thermosel System (Model 106) for elevated temperature testing. The thermal stabilities of the lubricants were studied using a TA Instruments TGA-2950 at a 10 °C/min heating rate in a nitrogen atmosphere (flow rate of 20 mL/min).

### 3. Results

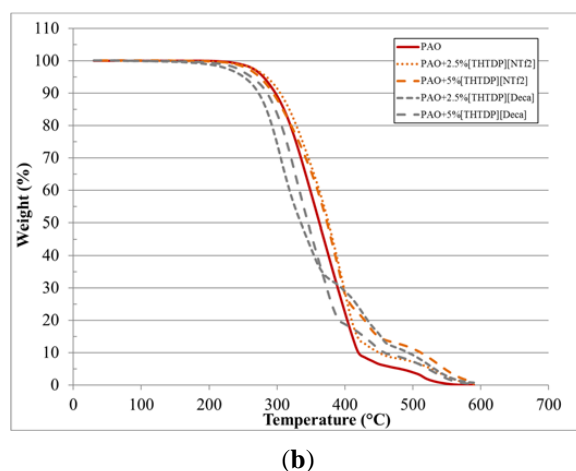
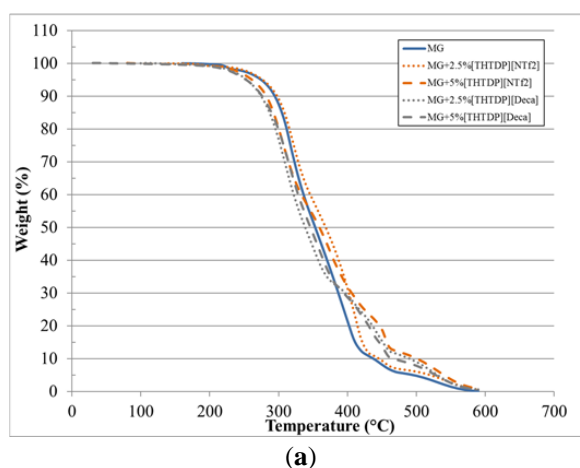
#### 3.1. Viscosity, Density, and Thermal Stability of the Lubricants

The density at 23 °C and dynamic viscosity at 40 °C and 100 °C of the lubricants used in this study are listed in Table 3. PAO and MG have similar densities at 23 °C. While adding [THTDP][NTf2] did not have any effect on the density of the base oils, the addition of [THTDP][Deca] slightly increased the density of the mixtures. Additionally, as can be seen in Table 3, PAO is a little more viscous than MG. The addition of ILs at such low concentrations caused little change in both oil viscosities.

**Table 3.** Density and viscosity values of the lubricants (\* Viscosity at 70 °C).

Lubricant	Density at 20 °C (g/cm <sup>3</sup> )	Dynamic Viscosity (cP)	
		40 °C	100 °C
MG	0.86	284	31
PAO	0.84	331	32
[THTDP][Deca]	0.88	1600 *	17
[THTDP][NTf2]	1.07	141	17
MG + 2.5%[THTDP][Deca]	0.86	285	31
MG + 5%[THTDP][Deca]	0.86	297	32
MG + 2.5%[THTDP][NTf2]	0.87	288	32
MG + 5%[THTDP][NTf2]	0.87	290	32
PAO + 2.5%[THTDP][Deca]	0.84	343	32
PAO + 5%[THTDP][Deca]	0.84	359	32
PAO + 2.5%[THTDP][NTf2]	0.85	332	32
PAO + 5%[THTDP][NTf2]	0.85	332	32

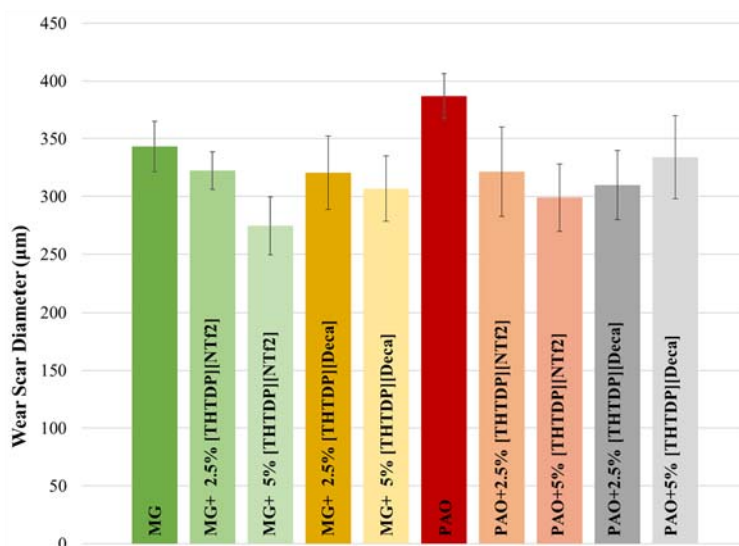
The thermogravimetric analysis (TGA) curves of the lubricants are shown in Figure 2. The onset decomposition temperatures for MG and PAO are 291 and 295 °C, respectively. The addition of the ILs, in most cases, slightly decreased the thermal stability of the lubricants. In either case, degradation for the mixtures does not reach 1% weight loss until 270 °C.



**Figure 2.** Weight loss *vs.* temperature thermogravimetric analysis (TGA) curves for the lubricants: (a) Mobilgear SHC XMP 320 (MG) and MG mixtures with ILs; (b) Synton PAO-40 (PAO) and PAO mixtures with ILs.

### 3.2. Effect of Ionic Liquid

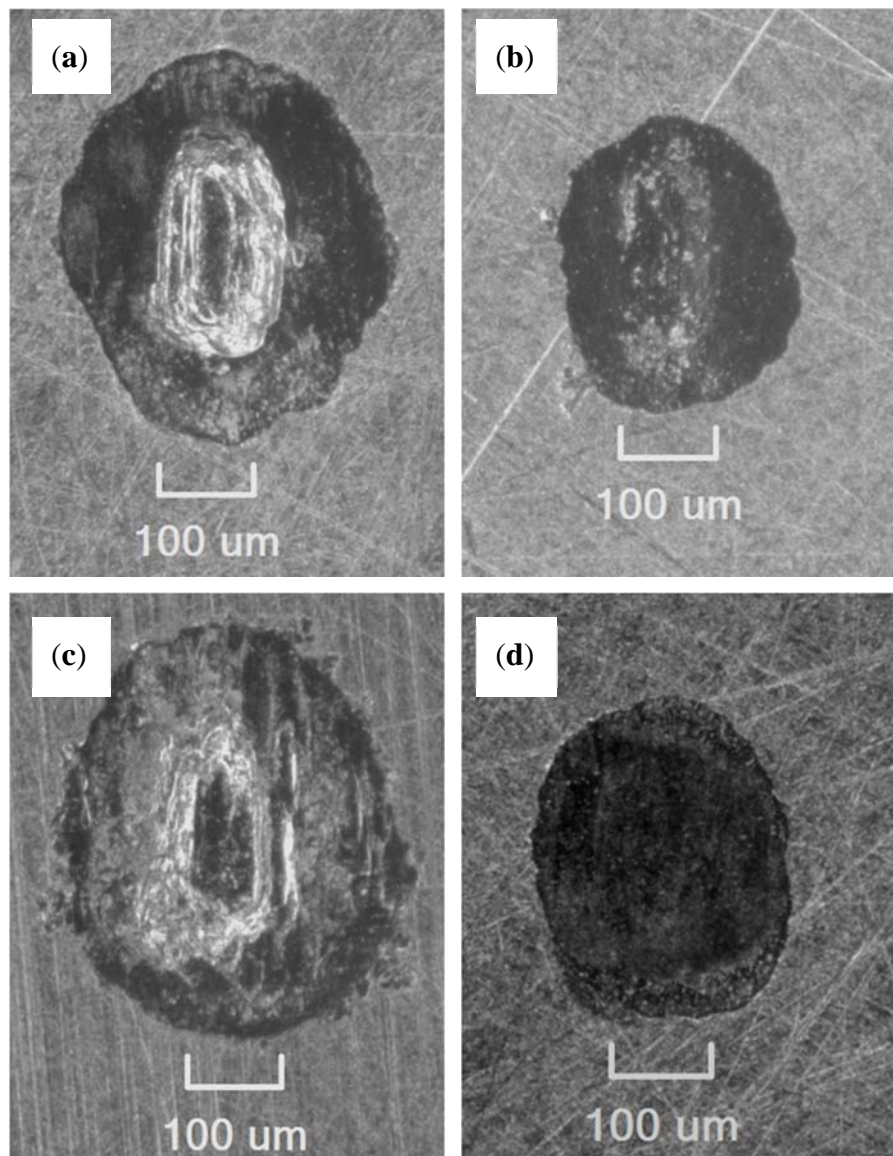
Figure 3 shows the wear scar diameters on AISI 52100 steel flat disks with normal ground finish ( $R_a \approx 0.1 \mu\text{m}$ ) after ball-on-flat reciprocating tests, using different lubricant mixtures. As expected, when PAO is used as lubricant, the steel disks presented higher wear than when commercially available MG is used. The reason for the larger diameters is the PAO does not contain any additive, while the additive package contained in the MG reduces friction and wear. Figure 3 also shows that adding ILs to MG and PAO reduced the wear scar diameter. Comparing the two ILs, the decrease in the wear scar diameter is greater for the [THTDP][NTf2], where a wear reduction of 20% and 23% is reached when 5 wt % of this IL is added to MG and PAO, respectively. In general, when [THTDP][NTf2] is used as an additive to both oils, increasing the ratio of the IL, wear of the steel disks decreased. However, if [THTDP][Deca] is added, the effect of increasing the IL ratio is opposite in both oils. A slight improvement of disk wear is observed when the ratio of [THTDP][Deca] is increased from 2.5 to 5 wt % in MG. However, when the same ratio of this IL is increased in PAO, the wear of the steel disk increased.



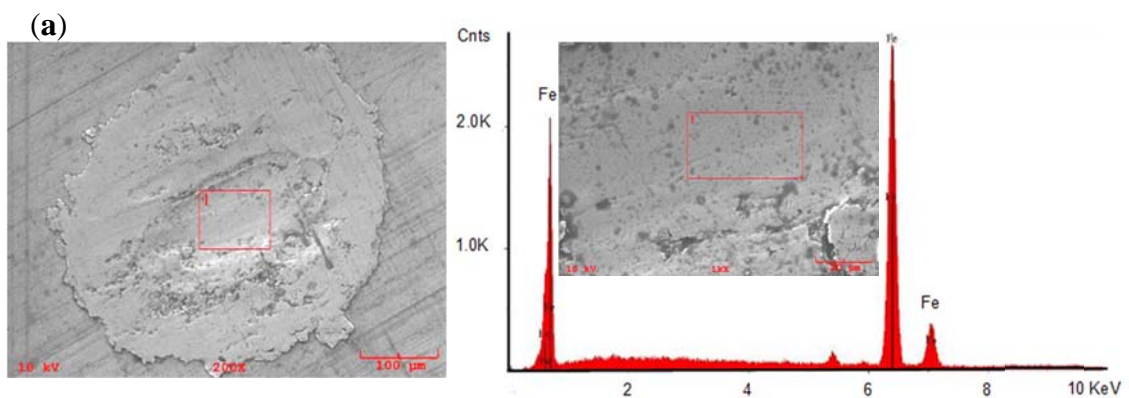
**Figure 3.** Wear scar diameters on AISI 52100 steel flat disks ( $R_a \approx 0.1 \mu\text{m}$ ) after ball-on-flat reciprocating tests.

Figure 4 shows wear scars on AISI 52100 steel flat disks ( $R_a \approx 0.1 \mu\text{m}$ ) after a ball-on-flat reciprocating test using MG, MG + 5% [THTDP][NTf2], PAO, and PAO + 5% [THTDP][NTf2] as lubricants. From the figure, the addition of the IL to the oils not only reduced the diameter of the wear scar, but also its depth. When the IL is not present, the worn surface presented severe grooving and plastic deformation, particularly for PAO (Figure 4c). The presence of cracks (Figure 5a) also confirms a fatigue component of the wear of the surfaces when oils without ILs are used. However, when [THTDP][NTf2] is added, a more superficial and smoother wear track is obtained, and the occurrence of fatigue cracks is reduced (Figure 5b). It has been reported [12–21] that ILs have the ability to form highly ordered absorbed layers on metal surfaces. This layer prevents direct contact between mating surfaces, reducing friction and wear. In this study, the presence of F and S on the worn surface (Figure 5b) after a test using [THTDP][NTf2] as an additive of PAO suggests the formation of a tribo-film on the steel disk surface that reduces the wear.

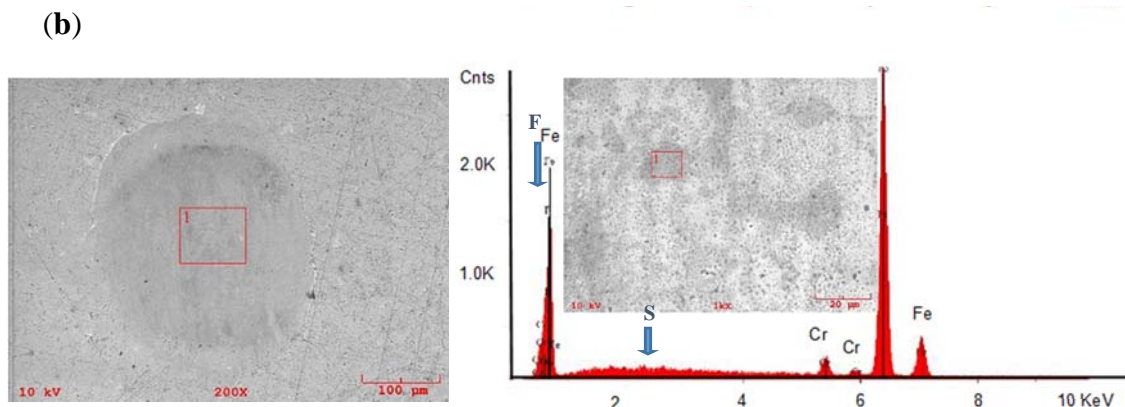




**Figure 4.** Wear scars on AISI 52100 steel flat disks ( $R_a \approx 0.1 \mu\text{m}$ ) after a ball-on-flat reciprocating test using the following lubricants: (a) MG; (b) MG + 5% [THTDP][NTf2]; (c) PAO; and (d) PAO + 5% [THTDP][NTf2].



**Figure 5.** Cont.

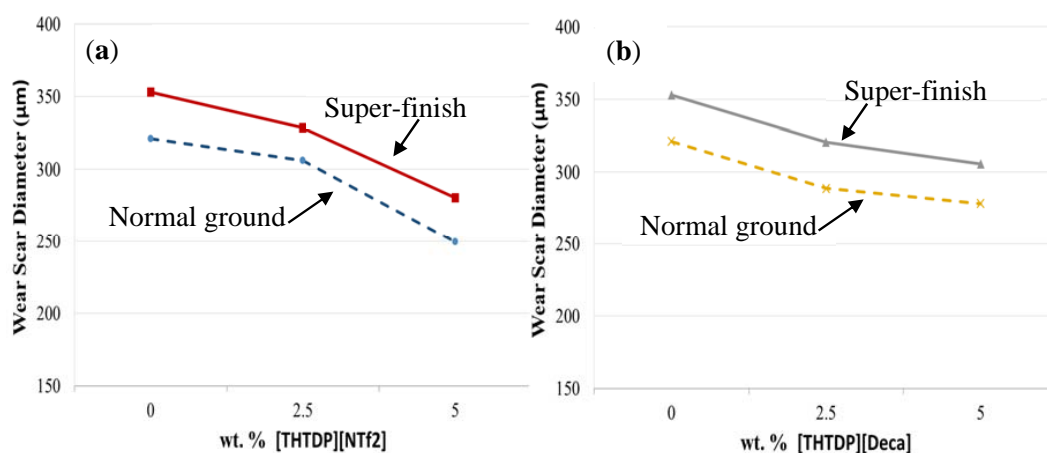


**Figure 5.** SEM micrographs and energy-dispersive X-ray spectroscopy (EDS) analysis of the wear scars on AISI 52100 steel flat disks ( $R_a \approx 0.1 \mu\text{m}$ ) after a ball-on-flat reciprocating test using the following lubricants: (a) PAO; (b) PAO + 5% [THTDP][NTf2].

### 3.3. Effect of Ionic Liquid

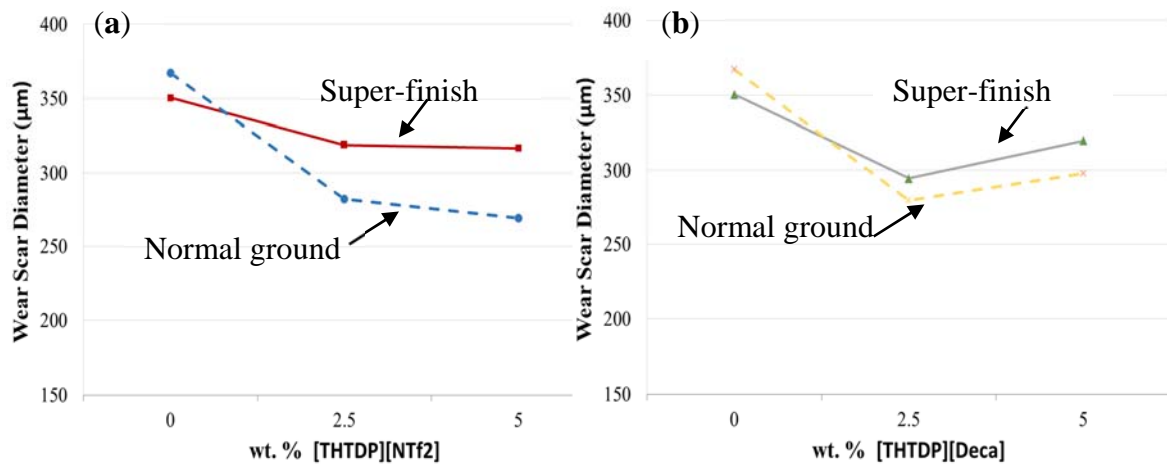
Figures 6 and 7 show the wear scar diameters on AISI 52100 steel flat disks for both surface finishes and both additives after a ball-on-flat reciprocating test using MG and PAO as base lubricants, respectively. The addition of ILs to both oils not only reduced the wear diameter on steel disks (Figures 6 and 7) but also smoothened the worn surface (Figure 8), resulting in a much milder wear mechanism. When MG was used as the base oil of the mixtures (Figure 6), increasing both IL concentrations resulted in a decrease in wear. However, as mentioned in Section 3.1, increasing the [THTDP][Deca] ratio in PAO (Figure 7) had a negative effect on the wear of the disk for both surface finishes.

In addition, studies have shown that super-finishing the working surfaces of mechanical components can reduce friction, pitting fatigue and wear [22,23]. As can be seen in Figures 6 and 7 only when a base oil is used with no additives, super-finishing the surface of the steel disk had a positive effect on wear. When the lubricant contains additives, *i.e.*, either ILs or additives present in the MG formulation, improving the surface finish ( $R_a \approx 0.02 \mu\text{m}$ ) of the disk produces larger wear tracks (Figure 8) than those obtained with a normal finish ( $R_a \approx 0.1 \mu\text{m}$ ) on the surface. This suggests that additives need a certain level of asperities to form a tribo-layer that improves the wear properties of the metals.

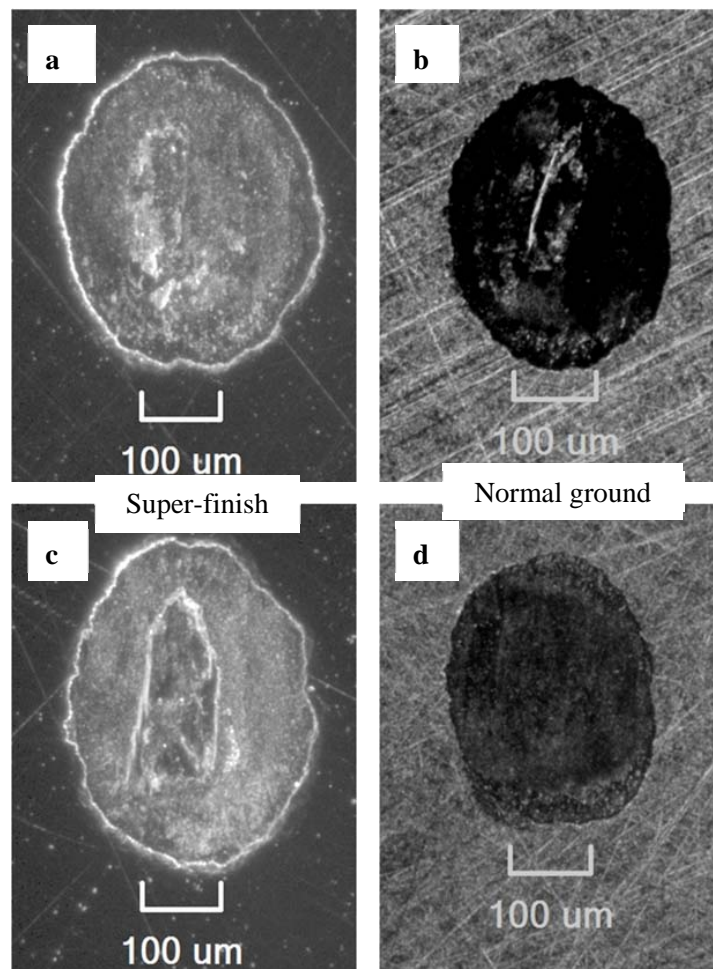


**Figure 6.** Wear scar diameters on AISI 52100 steel flat disks for both surface finishes after a ball-on-flat reciprocating test using MG as base lubricant and (a) [THTDP][NTf2], and (b) [THTDP][Deca] as an additive.





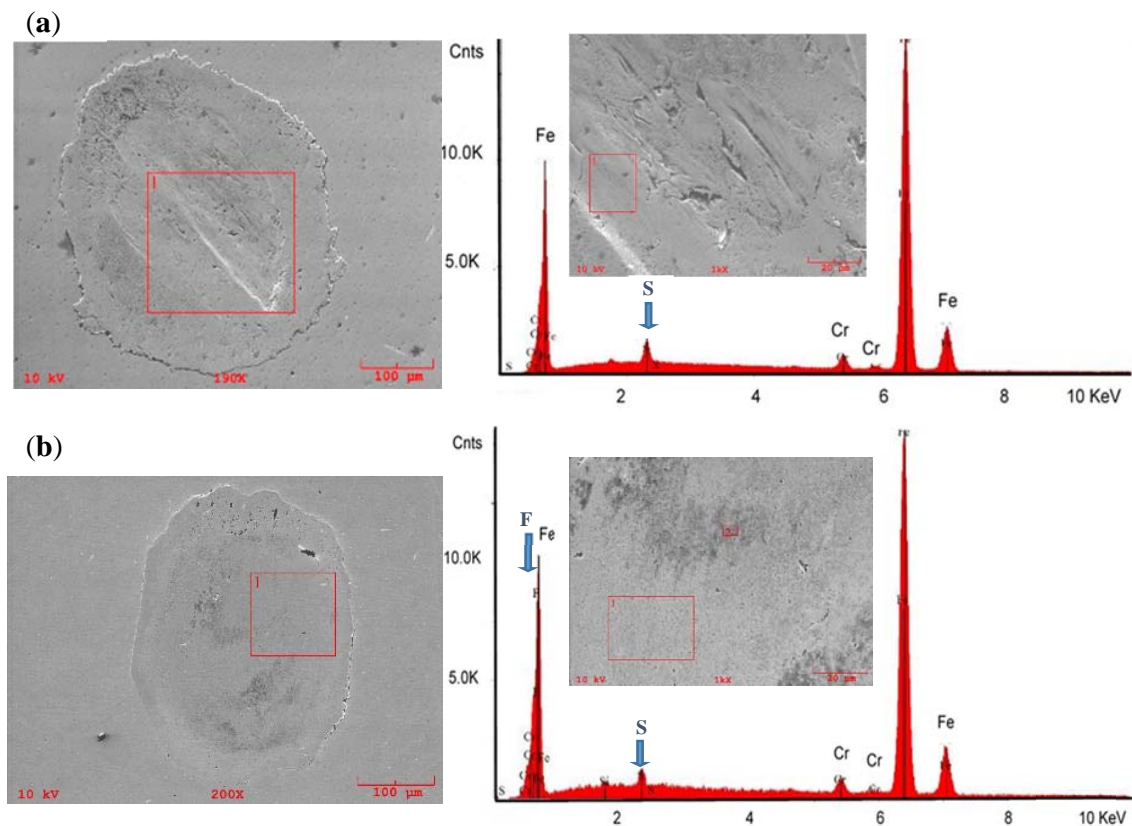
**Figure 7.** Wear scar diameters on AISI 52100 steel flat disks for both surface finishes after a ball-on-flat reciprocating test using PAO as base lubricant and (a) [THTDP][NTf2], and (b) [THTDP][Deca] as an additive.



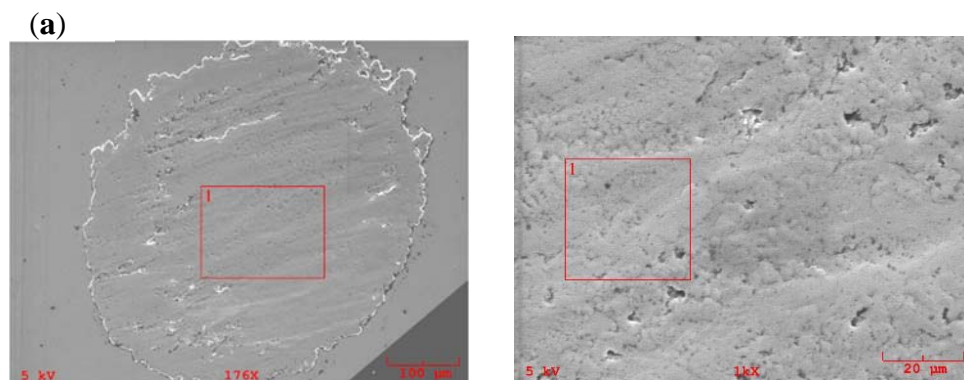
**Figure 8.** Wear scars on AISI 52100 steel flat disks after a ball-on-flat reciprocating test using: (a) and (b) MG + 5% [THTDP][Deca]; (c) and (d) PAO + 5% [THTDP][NTf2].

As can be seen in Figures 9 and 10 when MG and PAO are used without ILs, abrasive wear and plastic deformation, particularly for PAO (Figure 10), occurred. In addition, higher magnification

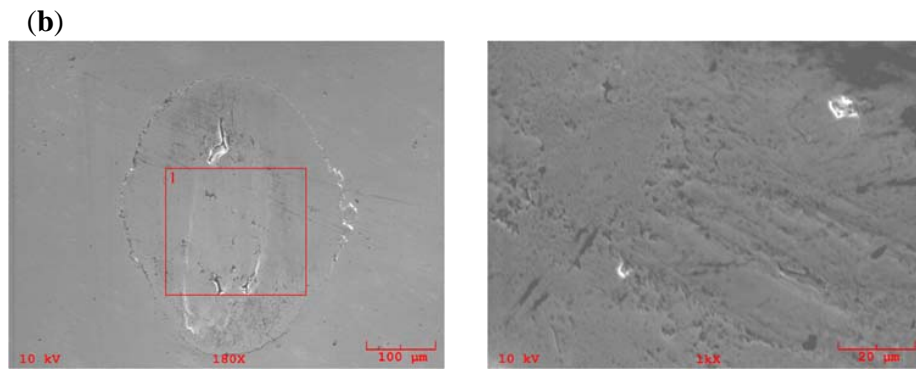
imaging of these worn surfaces showed that there was surface fatigue in these regions. Figure 9 also shows the EDS analysis of the wear scars on AISI 52100 steel flat disks ( $R_a \approx 0.02 \mu\text{m}$ ) after a reciprocating test using MG and MG + 5% [THTDP][NTf2]. The presence of sulfur on the worn surface suggests that this element, present in the MG formulation, reacted with the steel to form a sulfide layer [24] on the surface of the disk, reducing the worn area and the presence of cracks inside the wear track. The combination of [THTDP][NTf2] with the additives present in MG had a synergistic effect [15,25], producing a smoother and smaller wear track (Figure 9).



**Figure 9.** SEM micrographs and EDS analysis of the wear scars on AISI 52100 steel flat disks ( $R_a \approx 0.02 \mu\text{m}$ ) after a ball-on-flat reciprocating test using the following lubricants: (a) MG; (b) MG + 5% [THTDP][NTf2].

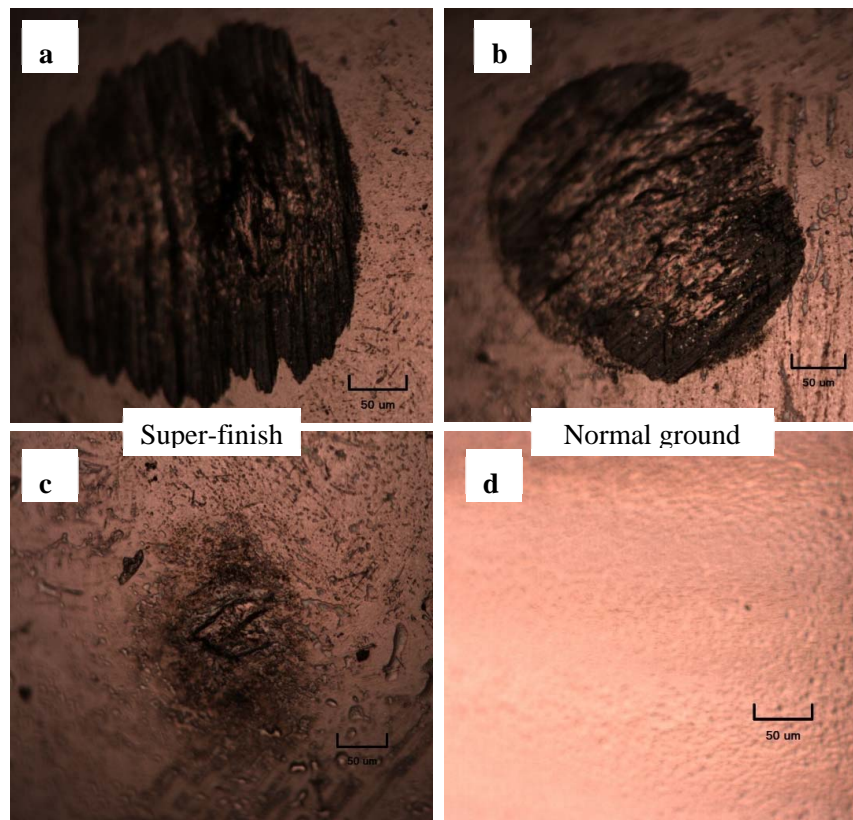


**Figure 10.** Cont.



**Figure 10.** SEM micrographs of the wear scars on AISI 52100 steel flat disks ( $R_a \approx 0.02 \mu\text{m}$ ) after a ball-on-flat reciprocating test using the following lubricants: (a) PAO; (b) PAO + 5% [THTDP][NTf2].

It should also be noted that, under the experimental conditions studied, the AISI 440C steel balls presented no apparent wear loss, but showed an adhered layer of steel particles from the disk (Figure 11) when IL was not present in the lubricant.



**Figure 11.** Optical micrographs of steel balls after a test lubricated with: (a) and (b) PAO; (c) and (d) PAO + 5% [THTDP][NTf2].

#### 4. Conclusions

This work investigates the potential tribological benefits of the use of two phosphonium-based ILs as additives of lubricants, in combination with two surface finishes, on gearboxes of both land-based and off-shore wind turbines.

Adding ILs to MG and PAO reduced the wear scar diameter for both surface finishes. This decrease is particularly important for [THTDP][NTf2], where a wear reduction of the steel disk

( $R_a \approx 0.1 \mu\text{m}$ ) around 20% and 23% is reached when 5 wt % of this IL is added to MG and PAO, respectively. When MG is used as the base oil of the mixtures, increasing both IL concentrations results in a decrease in wear. However, increasing the [THTDP][Deca] ratio in PAO has a negative effect on the wear of the disk for both surface finishes.

When the lubricant contains additives, *i.e.*, either ILs or additives present in the MG formulation, improving the surface finish of the disk produces larger wear tracks than those obtained with a normal finish on the surface. This suggests that additives need a certain level of asperities to form a tribo-layer that improves the wear properties of the metals.

When the IL is not present, the worn surface presented severe grooving and plastic deformation, particularly for PAO. In addition, the presence of cracks confirms a fatigue component of the wear of the surfaces when oils without ILs are used.

The presence of F and S on the worn surface after a test using [THTDP][NTf2] as an additive of PAO suggests the formation of a tribo-film on the steel disk surface that reduces the wear. The combination of [THTDP][NTf2] with the additives present in MG had a synergistic effect producing a smoother and smaller wear track.

**Acknowledgments:** Funding provided by the NYS Pollution Prevention Institute through a grant from the NYS Department of Environmental Conservation. Any opinions, findings, conclusions, or recommendations expressed are those of the author(s) and do not necessarily reflect the views of the Department of Environmental Conservation.

**Author Contributions:** Miguel A. Gutierrez performed the experiments under the supervision of Patricia Iglesias and Michael Haselkorn. All authors contributed equally to the design of the experiments and analysis of the data. Patricia Iglesias wrote the paper with cooperation of Michael Haselkorn.

**Conflicts of Interest:** The authors declare no conflict of interest.

## Abbreviations

The following abbreviations are used in this manuscript:

IL	Ionic Liquid
[THTDP][NTf2]	Trihexyltetradecylphosphonium bis(trifluoromethylsulfonyl) amide
[THTDP][Deca]	Trihexyltetradecylphosphonium decanoate
SEM	Scanning electron microscope
EDS	Energy-dispersive X-ray spectroscopy
PAO	Polyalphaolefin
TGA	Thermogravimetric analysis
MG	Mobilgear SHC XMP 320

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